

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization  
International Bureau



(43) International Publication Date  
30 May 2002 (30.05.2002)

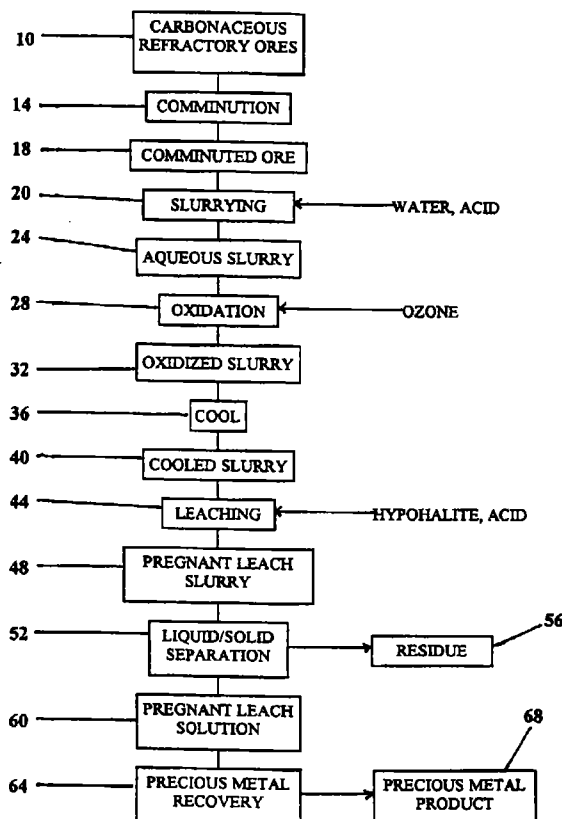
PCT

(10) International Publication Number  
**WO 02/42503 A1**

- (51) International Patent Classification<sup>7</sup>: C22B 3/06, 3/12, 11/00
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- (21) International Application Number: PCT/US01/43408
- (22) International Filing Date:  
21 November 2001 (21.11.2001)
- (25) Filing Language: English
- (26) Publication Language: English
- (30) Priority Data:  
60/252,271 21 November 2000 (21.11.2000) US
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- (81) Designated States (national): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.
- (84) Designated States (regional): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW),

[Continued on next page]

(54) Title: RECOVERY OF PRECIOUS METALS FROM CARBONACEOUS REFRACTORY ORES



(57) Abstract: In the process of the present invention, a carbonaceous refractory ore (10) is subjected to acidic oxidation (32) to destroy the carbonaceous compounds followed by acidic leach extraction (44) of the precious metal values.

WO 02/42503 A1



Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM),  
European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR,  
GB, GR, IE, IT, LU, MC, NL, PT, SE, TR), OAPI patent  
(BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR,  
NE, SN, TD, TG).

— *before the expiration of the time limit for amending the  
claims and to be republished in the event of receipt of  
amendments*

**Published:**

— *with international search report*

*For two-letter codes and other abbreviations, refer to the "Guid-  
ance Notes on Codes and Abbreviations" appearing at the begin-  
ning of each regular issue of the PCT Gazette.*

## RECOVERY OF PRECIOUS METALS FROM CARBONACEOUS REFRACTORY ORES

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### FIELD OF THE INVENTION

The present invention relates generally to processes for recovering precious metals from carbonaceous refractory ores and specifically to leaching processes for recovering precious metals from carbonaceous refractory ores.

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### SUMMARY OF THE INVENTION

The process for recovering precious metals from carbonaceous refractory materials, generally includes the following steps:

- (a) forming the carbonaceous refractory materials into an acid-containing feed slurry;
- 15 (b) contacting the feed slurry with an oxidizing agent to oxidize the carbonaceous material and form an oxidized slurry;
- (c) contacting the oxidized slurry with a hypohalite at a pH less than about pH 7 to dissolve the precious metal and form a pregnant leach slurry containing leached solids and a pregnant leach solution containing dissolved precious metals;
- 20 (d) separating the leached solids from the pregnant leach solution; and
- (e) recovering the precious metals from the pregnant leach solution.

The relatively simple and low cost process can recover precious metal, including gold, silver, platinum, and palladium, at relatively high rates. The process is more simplified and cost-effective than processes presently in use for the recovery of gold and  
25 other precious metals from carbonaceous ores, concentrates containing the same, or carbonaceous tailings. Consequently, the process can effectively treat precious metal-

containing materials that are too poor in recoverable metal values to be economically treated by conventional processes.

The feed slurry can contain a variety of acids, such as mineral acids. More preferably, the acid is selected from the group consisting of sulfuric acid, hydrochloric acid, nitric acid, acetic acid, phosphoric acid, hydrofluoric acid, hydrobromic acid, oxalic acid, and mixtures thereof, with mineral acids such as sulfuric acid, hydrochloric acid, and nitric acid being most preferred. Because the process uses such acids to dissolve precious metal values, it can avoid or minimize the environmental pollution often associated with conventional hydrometallurgical processes, which utilize environmentally harmful chemical compounds such as cyanide and thiourea in precious metal recovery.

In the contacting step (b), the carbonaceous material can be oxidized by a number of different oxidizing agents, such as ozone, molecular oxygen, chlorine gas, sulfuric acid, and alkaline hypochlorite, with ozone being most preferred. The slurry conditions are important to the effective oxidation of the carbonaceous material in the feed slurry. Preferably, the feed slurry in the contacting step (b) has a temperature ranging from about 40 to about 65 C, and the pH in the contacting step (b) ranges from about pH 1 to about pH3. When the oxidizing agent is ozone, the ozone is preferably contacted with the feed slurry at a rate ranging from about 0.25 to about 0.60 g/hr.

In the contacting or leaching step (c), the oxidized slurry is contacted with a hypohalite under acidic conditions to dissolve the precious metal. Preferably, the hypohalite is a hypohalite of alkali or alkaline earth metals. Surprisingly, it has been discovered that the hypohalite is more effective in dissolving precious metals in an acidic environment than in an alkaline environment. The most preferred hypohalites include

sodium hypochlorite, potassium hypobromite, calcium hypochlorite, potassium hypochlorite, calcium hypobromite, barium hypochlorite, barium hypobromite, and mixtures thereof.

The effectiveness of the hypohalite and acid in dissolving the precious metal values largely depend upon the concentration of the hypohalite and the pH of the slurry. Preferably, the concentration of the hypohalite in the pregnant leach slurry ranges from about 0.04 to about 0.4% by weight and the pH of the slurry ranges from about pH 2.2 to about pH 6.8.

The synergistic combination of the hypohalite and the acid provide an economical process by which gold-bearing sulfide ores that have been pretreated with such conventional techniques as autoclave oxidation and bio-oxidation can be subjected to direct application for the extraction of gold from the oxidized slurry. This would eliminate the expensive practice whereby gold-bearing sulfide ores pretreated in acidic solution by those oxidation techniques are washed and the pH adjusted to a high alkaline condition prior to applying conventional cyanidation.

#### DESCRIPTION OF DRAWINGS

The figure presents a preferred embodiment of a process according to the present invention.

#### DETAILED DESCRIPTION

The present invention relates to a process whereby improved recovery of gold is made from gold-bearing carbonaceous ores and other materials containing the same.

In accordance with the process of the invention, an acidified brine slurry of carbonaceous ores is treated with an effective quantity of ozone at ambient temperature which will convert the carbonaceous matter to an oxidized form that will enable gold and other metal values associated with the ore matrix to be solubilized in subsequent hydrometallurgical extraction step without interference by the carbonaceous impurities.

In a preferred embodiment of the invention, the aqueous slurry of carbonaceous ores mixed with a calculated amount of halide of alkali metals or alkali earth metals, generally in the range of 0.2 to 2.0 weight %, is acidified with any of the mineral acids such as sulfuric, hydrochloric, or nitric acid to a pH range between 1.0 and 3.0, with a preferred embodiment being 1.2 and 2.5. While the acidic ore slurry is thoroughly agitated by mechanical means and heated to a temperature in the range between 40° and 65°C, UV ozone from a commercial ozone generation source is dispersed into the slurry by a sparging action.

The slurry in the reaction vessel is cooled to room temperature upon completion of oxidation by the ozone treatment and the hydrometallurgical gold extraction follows. While agitation is still continued throughout the process, the oxidized ore slurry is mixed with an adequate amount of hypohalite of alkali metals or alkali earth metals such as sodium hypochlorite, potassium hypobromite, or calcium hypochlorite. At this time, the acidity of the slurry is measured and adjusted to an optimum pH range between 2.2 and 6.8, with the same acid initially used for the acidification in the ozone treatment step. It will normally take a period of time ranging from 2 to 10 hours, mostly from 4 to 8 hours, for a complete extraction of the metal values from the oxidized ore body using such as selected leaching chemicals. The recovery of gold from the pregnant solution is made by

any of those conventional techniques such as carbon-in-leach (CIL), carbon-in-pulp (CIP), or Merrill-Crowe precipitation process using zinc or aluminum.

Referring to the Figure, a preferred embodiment of the process of the present invention will now be described. Gold-bearing carbonaceous refractory ore 10 used for treatment pursuant to the present invention is crushed and pulverized 14 to produce a comminuted ore 18 having particle sizes to a maximum 100 mesh or particle diameter of less than 150 micrometers.

The detailed description will be made for this invention with reference to the following two major relevant steps: (1) conversion of carbonaceous matter admixed as impurities in the ore body into an oxidized form and (2) subsequent extraction of the metal values from the oxidized ore.

In slurring step 20, the pulverized (or comminuted) ore 14 of known weight is initially mixed in an open vessel containing an acidified brine solution and slowly, yet thoroughly agitated by means of an impeller system at speeds ranging from 100 to 500 rotations per minute to form an aqueous slurry 24. The density of aqueous slurry 24 normally comprises from 20 to 50 weight percent solids.

In the next step, the aqueous slurry 24 is oxidized 28 with ozone to form an oxidized slurry 32 in which the carbonaceous compounds are oxidized. To perform oxidation, the aqueous slurry 24 is continuously agitated at ambient temperature, while UV ozone from a commercial ozone generation source is sparged into the slurry at flow rates ranging from 0.25g to 0.60g per hour until completion of the oxidation reaction. This will take a total consumption of ozone in the range between 2.4g and 4.0g for the particular carbonaceous ore used in the present invention. However, the quantity of

ozone necessary for the complete oxidation will vary depending on the magnitude of carbonaceous impurities loaded in the ore matrix given the chemical reaction condition applied. The pH of the slurry during the ozone treatment will be maintained at about 1.0 to 3.0.

5        Upon completion of the ozone pretreatment, the oxidized slurry 32 in the processing vessel is cooled 36 to room temperature to form a cooled slurry 40.

      The cooled slurry 40 is leached 44 to form a pregnant leach slurry 48 including dissolved precious metals. In the leaching step 44, the agitation is continued for the next period of time ranging from 2 to 10 hours, and gold is extracted from the oxidized ore  
10    slurry by adding a selected hypohalite such as sodium hypochlorite as a leaching chemical in the concentration range of about 0.04 to 0.4%. After adding the leaching agent, the acidity of the cooled slurry 40 in the processing vessel is adjusted to a pH range between 2.2 and 6.8, and in general practice, the working pH range will be between 2.5 and 5.5, with a preferred embodiment being between 2.8 and 3.6. In most cases,  
15    however, the pH adjustment at this step is unnecessary because the acidity maintained during the ozone pretreatment will give a desirable pH range for leaching out the metal values in the subsequent step.

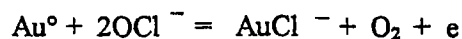
      It has been discovered that the recoverability of gold from carbonaceous refractory ores can be substantially improved by the acid-ozone pretreatment followed by  
20    conventional cyanidation techniques that involve filtering the oxidized slurry after the ozone treatment, adjusting the pH with lime, and leaching the metal out of the slurry with sodium cyanide for 24 hours. The ozone treatment has also considerably improved the recovery of precious metals including gold and silver from refractory ores. In one



process, the ore slurry is acidified to a pH level of 3.0 or below using preferably acetic acid is subjected to UV ozone treatment. After the ore matrix expands due to the ozone exposure, the pH level of the slurry is raised to pH 11.0 or higher using lime and caustic soda in order to leach out the precious metals. The highly alkaline ore slurry is then  
5 heated for a sufficient period of time at a preferable temperature of 180°F, while ozone has been continuously sparging through the slurry throughout the process to this point. The precious metals are leached out of the suitably basic slurry using leach chemicals such as cyanide and hypochlorite.

Contrary to these processes, in which the leach chemistry is realized in a basic  
10 solution on precious metals which have been oxidized, I have found that the acidic leaching process of the present invention is not only highly potent for the efficient recovery of gold as shown in the following examples, but it is also effective in recovering some members of platinum group metals such platinum and palladium. I have also found that there is no noticeable decrease in the leaching efficiency even at a temperature range  
15 as low as a few degrees above the freezing point by the present acidic leaching process.

The hypohalite ions such as those from sodium hypochlorite are good oxidizing agents, especially in acid solution, and the halite ions have the ability to function as ligands and form stable complexes with such metals as gold as  $\text{AuCl}_2^-$ ,  $\text{AuBr}_2^-$ , etc.;



20 Although it can be true in some applications that the hypohalite ions tend to decompose somewhat rapidly under the acidic condition, the metal complexes formed in the oxidation-reduction reaction in acid solution appear to be stable for an extended period of time. For instance, in one embodiment of the present invention, sodium

hypochlorite was used as a leach chemical for the extraction of gold from the slurry. At the end of the leaching step, the clean pregnant liquid containing the chloro-auroic complex was separated from the spent slurry by filtration, and the liquid was analyzed daily for gold for a period of two weeks. The results showed that the metal complex was  
5 stable for more than a week without a noticeable degree of decomposition. In basic solution, however, the hypohalite ions have the tendency to undergo disproportionation reaction, resulting in the production of halite ions as:



Therefore, the hypohalite ions will considerably lose the oxidizing power as well as the  
10 ability to complex with the precious metals.

The recovery of gold and other precious metals from the resulting pregnant liquid is made by performing a liquid/solid separation 52 to form a residue 56 and a pregnant leach solution 60 which is then subjected to precious metal recovery 64 to form a precious metal product 68. Precious metal recovery 64 can be performed by applying  
15 such techniques as carbon adsorption and Merrill-Crowe precipitation. It is also found that the metals can be recovered effectively in the acidic solution by precipitating them with aluminum. For instance, in one embodiment of the present invention, a series of 500 ml Erlenmeyer flasks are filled with clean pregnant liquid which has been produced from the leaching process. After adding aliquots of aluminum dust, the flasks are then  
20 magnetically agitated for 4 to 24 hours. Prior to the addition of aluminum dust, the pH of the leachate in the flasks ranges between 3.0 and 6.0. At the end of agitation, the clean spent solution from each flask is sampled for a quantitative determination of soluble gold still remaining in solution. This quantitative analysis is made by atomic absorption

spectrometry equipped with an electro-thermal atomization system. The results show that aluminum dust is able to precipitate gold greater than 99.9% in all replicate tests.

The following results from case studies are presented to further illustrate the process of the present invention. These examples are not intended by any means to be  
5 limiting the scope of this invention.

#### EXAMPLE 1

100 grams each of a hematitic oxide ore containing 0.19<sup>oz</sup>/<sub>t</sub> gold was placed in three replicate vessels and slurried with 300 ml of approximately 2.0% sodium chloride  
10 solution. While the processing vessels were agitated, the ore slurries were acidified with sulfuric acid to a pH of about 3.0 after mixing them with sodium hypochlorite. The agitation continued for seven hours to complete gold extraction, and the ore slurries were then filtered. Both the tailings and the pregnant liquid from three vessels were assayed for gold and the average value of gold extracted was calculated to be 94.3%.

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#### EXAMPLE 2

For a purpose of comparing the extractability of gold, the ore used in Example 1 was subjected to a cyanide treatment. Three replicate vessels, each charged with 100 grams of the oxide ore, were slurried with 300 ml of 0.1% sodium cyanide solution at a  
20 pH of about 11.5. The processing vessels were agitated for 24 hours and the slurries were filtered. Gold values were determined in the clean leachate as well as in the tailings. The data showed that an average of 90.2% total gold was extracted by cyanidation, as compared to 94.3% extraction by the method of this invention (see Example 1).

### EXAMPLE 3

A carbonaceous ore containing 0.32<sup>oz</sup>/t gold, and 3.4% by weight total carbon including 0.25% organic carbon was processed for the extraction of gold by the process of the present invention in parallel with standard cyanide leaching technique for  
5 comparison.

450 grams of the ore placed in a processing tank was slurried with 1.5L of the acidified brine solution and agitated. While the ore slurry was continuously agitated at ambient temperature, UV ozone was sparged into the slurry until the organic carbon impurities were eliminated by oxidation. The oxidized ore slurry was then mixed with  
10 leaching chemicals such as sodium hypochlorite and the pH of the slurry adjusted to approximately 3.0, if necessary. The agitation was further continued for the next 6 to 8 hours in order to leach gold out of the oxidized ore. In another leaching tank, 400 grams of the same ore was slurried with 1.2L of the cyanide solution used in Example 2, and agitated for 24 hours. After completion of the leaching process in both tanks, gold was  
15 determined in each pregnant solution and tailings. The result showed that 97.4% of the total gold was extracted from the ozone-oxidized ore, while 48.2% extraction was achieved by the cyanide leaching technique.

### EXAMPLE 4

20 The procedure of Example 3 was repeated for carbonaceous ore containing 0.13<sup>oz</sup>/t gold and 4.98% total carbon including 0.49% by weight organic carbon impurities. Gold recovery was 97.7% by the process of the present invention as compared to only 41.6% recovery by conventional cyanide leaching.

The results shown in the examples above illustrate that the recovery of gold from carbonaceous ores is substantially improved by the acid-ozone pretreatment in combination with the acidic hypohalite leaching according to the present invention. It is also clearly indicated that the acidic hypohalite leaching of the present invention for gold  
5 recovery from the oxidized ores is highly effective and superior to the conventional cyanide leaching technique, not only in terms of the potency of the metal extraction, but also in terms of shorter leaching time.

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What is claimed is:

1. A method for recovering precious metals from carbonaceous refractory materials, comprising:
  - (a) forming the carbonaceous refractory materials into a feed slurry, the feed  
5 slurry comprising an acid;
  - (b) contacting the feed slurry with an oxidizing agent to oxidize the carbonaceous material and form an oxidized slurry;
  - (c) contacting the oxidized slurry with a hypohalite of alkali metals or alkali earth metals at a pH less than about pH 7 to dissolve the precious metal and form a  
10 pregnant leach slurry containing leached solids and a pregnant leach solution containing dissolved precious metals;
  - (d) separating the leached solids from the pregnant leach solution; and
  - (e) recovering the precious metals from the pregnant leach solution.
2. The method of Claim 1, wherein the precious metal is selected from the  
15 group consisting of gold, silver, platinum, palladium, and mixtures thereof.
3. The method of Claim 1, wherein the acid is selected from the group consisting of sulfuric acid, hydrochloric acid, nitric acid, acetic acid, phosphoric acid, hydrofluoric acid, hydrobromic acid, oxalic acid, and mixtures thereof.
4. The method of Claim 1, wherein the feed slurry in the contacting step (b) has  
20 a temperature ranging from about 40 to about 65 C.
5. The method of Claim 1, wherein the oxidizing agent is selected from the group consisting of ozone, molecular oxygen, chlorine gas, sulfuric acid, alkaline hypochlorite, and mixtures thereof.

6. The method of Claim 1, wherein the pH in the contacting step (b) ranges from about pH 1 to about pH 3.

7. The method of Claim 1, wherein the oxidizing agent is ozone and the ozone is contacted with the feed slurry at a rate ranging from about 0.25 to about 0.60g/hr.

5 8. The method of Claim 1, wherein the hypohalite is selected from the group consisting of sodium hypochlorite, potassium hypobromite, calcium hypochlorite, potassium hypochlorite, calcium hypobromite, barium hypochlorite, barium hypobromite, and mixtures thereof.

9. The method of Claim 1, wherein the concentration of the hypohalite in the pregnant leach slurry ranges from about 0.04 to about 0.4% by weight.

10 10. The method of Claim 1, wherein the pH in the contacting step (c) ranges from about pH 2.2 to about pH 6.8.

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11. A method for recovering precious metals from carbonaceous refractory materials, comprising:

(a) forming the carbonaceous refractory materials into a feed slurry, the feed slurry comprising an acid;

5 (b) contacting the feed slurry with a hypohalite of alkali metals or alkaline earth metals at a pH less than about pH 7 to dissolve the precious metal and form a pregnant leach slurry containing leached solids and a pregnant leach solution containing dissolved precious metals;

(c) separating the leached solids from the pregnant leach solution; and

10 (d) recovering the precious metals from the pregnant leach solution.

12. The process of Claim 11, further comprising contacting the feed slurry with an oxidizing agent to oxidize the carbonaceous material in the feed slurry.

13. The method of Claim 11, wherein the precious metal is selected from the group consisting of gold, silver, platinum, palladium, and mixtures thereof.

15 14. The method of Claim 11, wherein the acid is selected from the group consisting of sulfuric acid, hydrochloric acid, nitric acid, acetic acid, phosphoric acid, hydrofluoric acid, hydrobromic acid, oxalic acid, and mixtures thereof.

15. The method of Claim 12, wherein the feed slurry in the contacting step has a temperature ranging from about 40 to about 65 C.

20 16. The method of Claim 12, wherein the oxidizing agent is selected from the group consisting of ozone, molecular oxygen, chlorine gas, sulfuric acid, alkaline hypochlorite, and mixtures thereof.



17. The method of Claim 12, wherein the pH in the contacting step ranges from about pH 1 to about pH 3.

18. The method of Claim 12, wherein the oxidizing agent is ozone and the ozone is contacted with the feed slurry at a rate ranging from about 0.25 to about 0.60 g/hr.

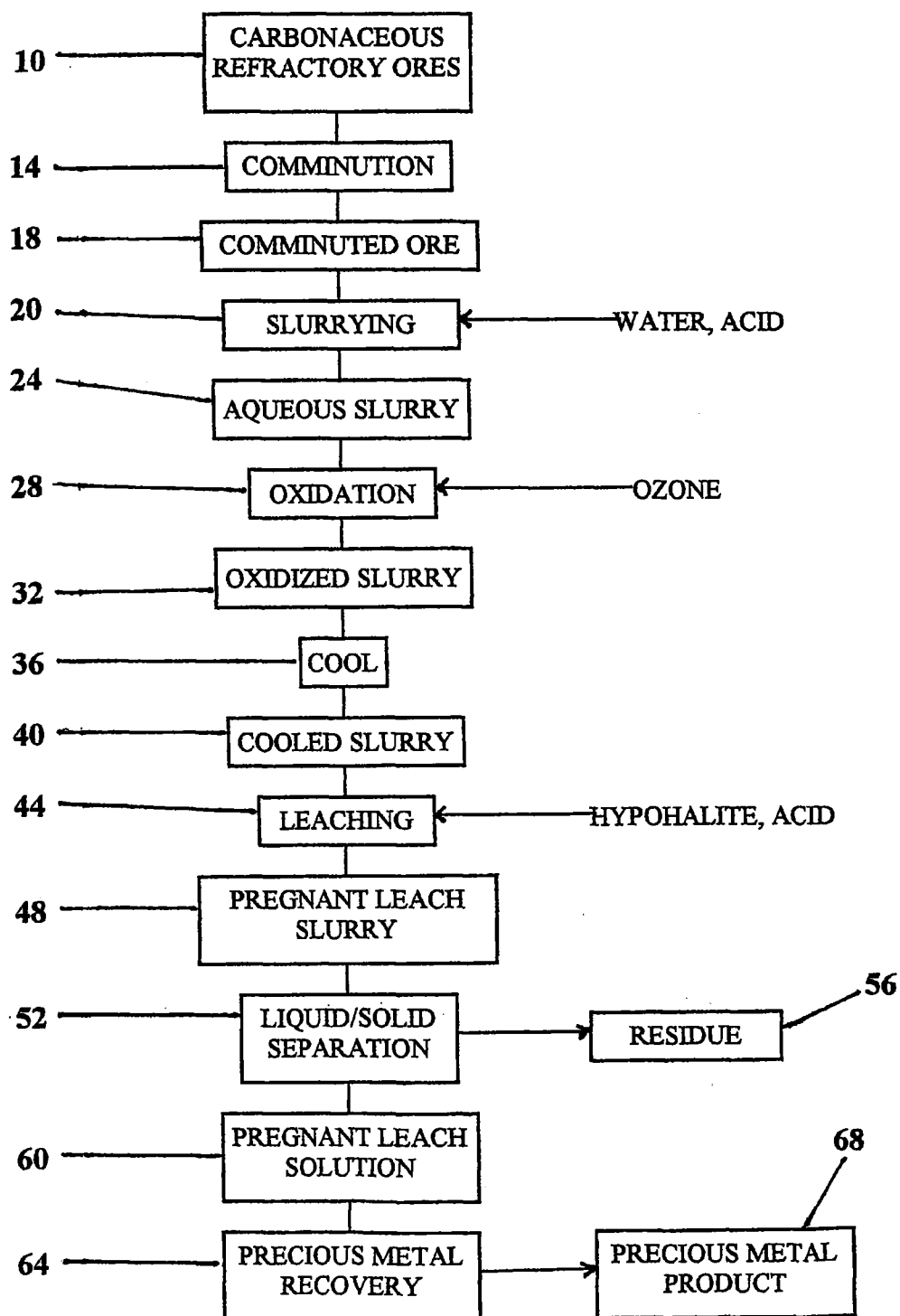
5        19. The method of Claim 12, wherein the hypohalite is selected from the group consisting of sodium hypochlorite, potassium hypobromite, calcium hypochlorite, potassium hypochlorite, barium hypochlorite, calcium hypobromite, barium hypobromite, and mixtures thereof.

20. The method of Claim 11, wherein the concentration of the hypohalite in the  
10 pregnant leach slurry ranges from about 0.04 to about 0.4% by weight.

21. The method of Claim 11, wherein the pH in the contacting step ranges from about pH 2.2 to about pH 6.8.

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**FIG.**

## INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US01/43408

**A. CLASSIFICATION OF SUBJECT MATTER**

IPC(7) : C22B 3/06, 3/12, 11/00

US CL : 75/744

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 75/744

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EAST  
hypohalite, hypochlorite, hypobromite

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4,979,986 A (HILL et al ) 25 December 1990 (25.12.2000), column 4, line 8 to column 6, line 56.	1-3, 5, 6, 8-14 16, 17, 19- 21
A	US 5,147,617 A (TOURO et al) 15 September 1992 (15.09.1992), claims.	1-21
Y	JP 6136465 A (KIKUCHI et al) 17 May 1994 (17.05.1994), abstract.	1-4, 11-15, 19
X	US 5,169,503 A (BAUGHMAN et al ) 08 December 1992 (08.12.1992), column 2, line 50 to column 4, line 23.	1-3, 5, 8, 10-13, 16, 19, 21
A	DERWENT ACC. NO. 1993-000460 for CS 90066696 AS (DOLEZEL et al) 15 July 1992 (15.07.1992), see abstract.	1-21

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
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"P" document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search  
15 MARCH 2002

Date of mailing of the international search report  
**29 MAR 2002**

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